



Short Note

Determination of Harvest Time Residues of Thiacloprid in Cardamom

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Cardamom, *Elettaria cardamomum* (L.) Maton. the queen of spices is indigenous to the Southern states of India. It is cultivated in Western Ghats (Kerala, Tamil Nadu and Karnataka) in an area of 73,795 ha. with a production of 12,540 MT (2005-06) and one of the important products fetching enormous foreign exchange (Stanley, 2007). India was the world's largest producer and exporter until it was taken over by Guatemala in the 18th century. One of the major constraints in the production of cardamom is the excessive damage caused by pests. At present, these pests are kept under check with the help of synthetic insecticides. With the strict legislations enforced by the EPA, cardamom capsules with pesticide residues have a chance of being rejected by the hitherto importing countries, which in turn would have a major say in foreign revenues.

Keywords: Thiacloprid, harvest time residues, cardamom

The pesticide use pattern in the present day situations has led to resistance build-up by pests and pesticide residues, which demands newer and safer pesticides with different modes of action. Thus, there is a greater need to evaluate pesticides that would leave no or lesser residues in the commodity as well as in the environment. Chloronicotinylns/ neonicotinoids are the new group of crop protection agents highly effective against sucking pests which act on receptor protein of insect nervous system (Preetha, 2008). Thiacloprid, a new chloronicotinyl insecticide, is targeted to control sucking and biting insects in cotton, rice, vegetables, pome fruit, sugar beet, potatoes and ornamentals. It disrupts the nervous system by acting as an inhibitor at nicotinic acetylcholine receptors. Thiacloprid is slightly mobile in soil and hence it has no potential for leaching into ground water. Nowadays this pesticide used in the cardamom to control the thrips. Keeping all these in views, the present investigation was undertaken to determine the harvest time residues of thiacloprid in cardamom capsules.

Materials and Methods

Field experiment was conducted to determine the harvest time residues of thiacloprid on and in Green gold variety cardamom during May, 2006 in the farmer's holding at Madhavanganal near Kumily, Idukki district, Kerala. The experiments were conducted with three treatments viz., T₁ - Untreated control, T₂ - Thiacloprid 240 SC @ 25 g a.i. ha⁻¹ and T₃ - Thiacloprid 240 SC @ 50 g a.i. ha⁻¹. The crop was maintained properly by adopting standard agronomic practices recommended by Tamil Nadu

Agricultural University. The treatments were imposed when the pests crossed the economic threshold level (ETL). Three sprays were given with a pneumatic knapsack sprayer with a spray fluid volume of 500 litres ha⁻¹.

Sampling

Matured and uniform sized cardamom capsules were collected at random on 30 days after third spray with the help of forceps for residue analysis. From each plot, 150 g of green capsules was collected and from this, a sub sample of 20 g green capsules in duplicate was taken for fresh sample analysis and transferred immediately into the sample container with acetonitrile. The remaining sample of 100 g was divided into two portions and was cured under conventional curing chamber at maximum temperature of 60 - 65°C maintained for 24 h and used as cured samples for residue analysis. The weights of the samples before and after curing were recorded from each plot to workout the residues on moisture free basis and curing loss.

Extraction

The weighed sample of 20 g was soaked in acetonitrile (50 ml) overnight, homogenized and filtered through Buchner funnel. After repeated washing, the pooled acetonitrile extract was evaporated to near dryness and the residue was taken up in the 20 ml water.

ChemElut® column cleanup

The aqueous solution was placed on the top of the ChemElut® CE 1020 columns and allowed to

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the liquid in the column for about 15 min for equal distribution. The residues of thiacloprid were eluted with 50 ml of cyclohexane / ethylacetate (HPLC grade 1/1 v/v). The eluent was collected in a 250 ml round bottom flask and evaporated near dryness and residue was taken by dissolving in 2 ml HPLC grade ethylacetate.

Florilil column cleanup

Fifty ml of ethylacetate was allowed to drain through a chromatography column packed with 10 g of florilil sandwiched with anhydrous sodium sulphate. The residue eluted from ChemElut® columns was allowed on the florilil column at the flow rate of one milliliter per minute. Then the thiacloprid residue was eluted using 30 ml acetonitrile. The elutant was concentrated to near dryness, the residue dissolved in acetonitrile: water (1/1, v/v) and fed into HPLC.

Preparation of standards

The stock solution of 1000 ppm was prepared by dissolving 101 mg of thiacloprid technical material (99.0% purity) in 100 ml of acetonitrile (HPLC grade). From this stock, intermediate stock solutions of 100 and 10 ppm were prepared. Using 10 ppm stock, working standards of 0.5, 1, 2, 3, 5 and 10 ppm were prepared in HPLC grade acetonitrile.

Recovery studies

Samples were fortified with working standards at 0.1, 0.5 and 1.0 ppm level to find out the recovery of thiacloprid. The recovery obtained was 83.52 per cent.

Final quantification

End analysis was done with the aid of High Performance Liquid Chromatography (HPLC), Hitachi model L 6200 with the following operating parameters. Mobile phase : Acetonitrile (HPLC grade): Water (80:20 V/V); Column : ODS 2; Flow rate : 1 ml min⁻¹; Wave length : 270 nm; Quantity injected : 20 μ l (fixed loop). The amount of residue was determined by comparing the sample response with the response of standard by using the formula.

where, H_s - Peak height of the sample; H_{std} -

$$\text{Residues in ppm} = \frac{H_s}{H_{std}} \times \frac{W_{std}}{W_s} \times \frac{V_{ex}}{V_s} \times \frac{A_s}{A_{std}}$$

Peak height of the standard; W_{std} - Weight of the standard injected in ng; W_s - Weight of the sample in g; V_{ex} - Volume of the final extract in ml; V_s - Quantity of the sample injected in μ l; A_s - Attenuation of the sample; A_{std} - Attenuation of the standard

Results and Discussion

The mean recovery was 83.52 per cent from samples fortified at 0.1, 0.5 and 1.0 ppm level. Hence the recovery factor was not used for working out the residues. The minimum detection limit of the instrument was 0.5 ppm and the determinability level in the sample was 0.05 μ g g⁻¹ considering the weight of the sample as 20 g and final volume of the extract as 2 ml. The harvest time residues of thiacloprid

Table 1. Harvest time residues of thiacloprid 240 SC in/ on cardamom

Treatment	Dose (g a.i. ha ⁻¹)	Residues in μ g g ⁻¹ at harvest	
		Green capsules	Cured capsules
T ₁ - Untreated check	-	BDL	BDL
T ₂ Thiacloprid 240 SC	25	BDL	BDL
T ₃ Thiacloprid 240 SC	50	BDL	BDL

BDL- Below detectable level

240 SC at 25 and 50 g a.i. ha⁻¹ as foliar spray were at below detectable level (BDL) in green and cured cardamom capsules (Table 1). The interval between the last spray and sample picking was 30 days. Similar results were obtained by Stanley (2007), who reported that the residues of diafenthiuron dissipated to 0.08 and 0.16 per cent in green cardamom capsules at 15 DAT. Renuka (2001) and Rajabaskar (2003) reported a total loss of profenofos after 15 days of spray in both green and cured cardamom capsules when sprayed at 0.075 per cent. Picking of cardamom capsules was carried out at an interval of 30 - 35 days. As harvest being the focal point for enforcement of residue tolerances, the suggested waiting periods of seven days is safe enough to control the cardamom pests with thiacloprid without the problem of pesticide residues in harvestable produce.

References

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